भारतीय मानक **IS 5182 (Part 2/Sec 1) : 2023**

Indian Standard

वायु प्रदूषण को मापने के तरीके

भाग 2 सल्फर डाइऑक्साइड

अनुभाग 1 टेट्राक्लोरोमर्क्युरट/पैरारोसनीलिने विधि

(दूसरा पुनरीक्षण)

Methods for Measurement of Air Pollution

Part 2 Sulphur Dioxide

Section 1 Tetrachloromercurate/Pararosaniline Method

(Second Revision)

ICS 13.040.20

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June 2023 **Price Group 7**

Air Quality Sectional Committee, CHD 35

FOREWORD

This Indian Standard (Part 2/Sec 1) (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

Practically all fuels in common use contain variable amounts of sulphur, most of which is discharged to the atmosphere as sulphur dioxide during combustion. In addition, specific industrial process produces large quantities of sulphur dioxide, some of which may escape into the air. The concentration to be found in the atmosphere is governed not only by the number and size of local sources but by factors, such as the height of chimneys and meteorological conditions.

Pollution of the air by sulphur dioxide is widespread since it exists wherever fossil fuels are burned. Concentrations in the atmosphere are normally far below the level which can be detected by odour but, being always present to some extent, the gas does produce detectable effects. It is harmful to flora, fauna including human health, building, stone and metal work, it can kill useful micro-organism, and aggravate illnesses and ultimately deteriorate environment quality in total.

Because it is known to be potentially harmful for both environment and the economics, a knowledge of the level at which sulphur dioxide is present in the air is important. The prevailing trends of annual average concentration of SO_2 in India in metropolitan cities is reported to be varying from 5 µg/m^3 to 50 µg/m^3. Concentration of pollutants in air is also dependent on meteorological data with respect to temperature, relative humidity, rainfall on the day of sampling. The wind speed and mixing height data can also influence the level of concentrations.

This standard was published in 1969. The first revision was published in 2001, in which the sampling clause was modified to suit specific needs. Sampling efficiency was defined, in addition to defining the precision and accuracy of the method. Applicability, range and sensitivity and the possible interferences of the method were also identified.

In this second revision, the TCM method also called improved west and gaeke method has been updated based on the international standard ISO 6767 : 1990 'Ambient air — Determination of the mass concentration of sulfur dioxide — Tetrachloromercurate (TCM)/pararosaniline method' and studies done by National Environment Engineering Research Institute (CSIR-NEERI, Delhi) to reduce the use of mercury in preparation of potassium tetrachloromercurate. In addition, the sampling clause is further modified to help improve the field work as well as emphasis is laid on various quality assurance and quality control measures as calibration of sampler and intermediate checks, leak check, use of field blanks and certified reference material. An explanatory note on recovery of mercury from waste of absorbing reagent and disposal is as follows:

The use of mercury is reduced by 75 percent in this revision based on studies carried out by CSIR-NEERI Delhi. In addition, can collect the mercury containing solutions after test in a polyethylene container. After collecting 40 l mercury containing solution, neutralize with 0.4 percent Sodium Hydroxide (NaOH) solution with constant stirring using aerator. After neutralizing add 400 ml more 0.4 percent Sodium Hydroxide solution to the mercury containing solution. Add 100 gm Sodium Sulphide Nanohydrate ($Na_2S.9H_2O$) solution. Wait for 10 min to 15 min to complete the reaction. Add 400 ml of Hydrogen Peroxide Solution (30percent). Keep the container for 24 h to separate the residue and liquid. After 24 h remove the clear supernatant solution and discard it. Collect the residue having high concentration of mercury and dispose as per the relevant laws.

The procedure of calibration curve using sulphur dioxide permeation tubes has been removed due to issues in commercial availability of calibrated permeation tubes in country. The Committee has also decided to renumber this standard as IS 5182 (Part 2/Sec 1), since a new standard IS 5182 (Part 2/Sec 2) has been developed which deals with instrumental analysis of sulphur dioxide using UV fluorescence method.

In the preparation of this standard considerable assistance was derived from the following publications:

a) BS 1747-3 : 1969 Method for the measurement of air pollution — Part 3 Sulphur dioxide;

b) Report of the working party on methods of measuring air pollution and survey techniques 1964, a publication of Organization for Economic Co-operation and Development, Paris; and

c) Selected methods for the measurement of sulphur dioxide, a publication of US Department of Health, Education and Welfare, Public Health Service, USA.

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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 2 SULPHUR DIOXIDE

SECTION 1 TETRACHLOROMERCURATE/PARAROSANILINE METHOD

(Second Revision)

1 SCOPE

This standard (Part 2/Sec 1) prescribes the tetrachloromercurate/pararosaniline method for the measurement of concentration of sulphur dioxide (SO_2) present in the ambient air.

2 REFERENCE

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 2303 (Part 1/Sec 1) : 2021/ISO 719 : 2020	Grading glass for alkalinity: Part 1 hydrolytic resistance of glass grains, Section 1 Determination and classification of hydrolytic resistance at 98c (*third revision*)
IS 4167 : 2020	Glossary of terms relating to air pollution (*second revision*)
IS 5182 (Part 14) : 2000	Methods for measurement of air pollution Guidelines for planning the sampling of atmosphere

3 TERMINOLOGY

For this standard, the definitions given in IS 4167 shall apply.

4 PRINCIPLE

Sulphur dioxide is absorbed from air in a solution of potassium tetrachloromercurate (TCM). A dichlorosulphitomercurate complex which resists oxidation by the oxygen of the ambient air, is formed. The complex is made to react with pararosaniline in presence of formaldehyde to form para-rosaniline methyl sulphonic acid which give colour in the solution. Intensity of colour is directly proportional to the SO_2 present in the solution. The absorbance of the solution is measured at 560 nm by means of a suitable spectrophotometer.

5 RANGE AND SENSITIVITY

This is colorimetric method for measurement of sulphur dioxide in ambient air with accuracy of 10 percent up to SO_2 concentration of 200 µg/m^3. Accuracy improves further for higher concentration of SO_2.

Beer's Law is applicable for full working range of the method from 0.03 to 1.0 absorbance unit. This corresponds to 0.03 µg/ml to 1 µg/ml of sulphite ion as sulphur dioxide. The lower detection limit in ambient air is estimated to be 2.5 µg/m^3 for 24 h sampling with a flow rate of 0.5 litre/min. Higher concentrations can be analyzed by dilution (1 to 10 times).

6 INTERFERENCES

The effects of principal known interferences have been minimized or eliminated in this method. Interferences by oxides of nitrogen up to 5 µg/ml are eliminated by adding 1 ml of 0.6 per cent sulphamic acid at the time of analysis. Ozone is made to decompose by allowing the solution to stand for 20 min prior to analysis. The interference of trace metals is eliminated by the addition of ethylene diamine tetra acetic acid (EDTA) to the absorbing solution prior to sampling. At least 60 µg iron (III), 30 µg manganese (II) and 10 µg chromium (III) in 10 ml absorbing reagent do not create positive interfere. No significant interference was found from 10 µg copper (II) and 22 µg vanadium (V). Ammonia, sulphides, and aldehydes do not interfere in analysis.

7 APPARATUS

7.1 Gas sampler to absorb sulphur dioxide from air in impingers shall consist of the following:

7.1.1 *Cabinet* — The cabinet can be of the following two types:

a) Type-I: Cabinet with built in sampling pump; and
b) Type-II: Cabinet without sampling pump in which case the suction for sampling the gases is provided by the blower/pump of particulate sampler. Except the sampling pump, both Type-I/II cabinets should be designed to suitably hold items as listed from **7.1.1.1** to **7.1.1.8**.

7.1.1.1 Sampling pump whether built in or external should be capable of sucking unfiltered air and capable of maintaining flow rate of 10 litres/min free flow with pulse dampening system.

7.1.1.2 *Cold box*

A cold box capable of holding 4 impingers capable of thermoelectrically maintaining temperature of absorbing reagents in impingers during sampling at (15 ± 5) °C. This is required for optimum absorption of gases in absorbing reagent as well as to avoid loss of absorbing reagent by evaporation during sampling in the summer season. The cold box housing should be insulated and deep enough to cover the impingers up to the level of absorbing solution for effective temperature control around absorbing solutions.

7.1.1.3 The cabinet shall have a digital temperature indicator cum controller with a resolution of 0.1 °C and accuracy of ± 1 °C to continuously monitor and display the temperature of impinger sampling reagents.

7.1.1.4 *Arrangement for measurement, control and distribution of flow*

A rotameter shall be used for flow measurement with a range of 0 lpm to 1.5 lpm with resolution of 0.05 litre/min and accuracy of ± 2 percent full scale. The system will have four inlets individually connected to the outlet of each impinger on one end and other end to flow control valve via particulate trapping filters. Outlet of the manifold unit shall be connected to the suction pump. Material of control valves of the manifold should be made up of non-corrosive metal. Entire sampling line and a manifold shall be leakage free. Leak check system must be integral part of the sampling unit.

7.1.1.5 *Impingers*

All glass standard 35 ml midget impinger with dimensions as shown in Fig.1 shall be used for sampling. The glass impingers shall be made from class HGB 3 glass conforming to IS 2303 (Part l/Sec 1).

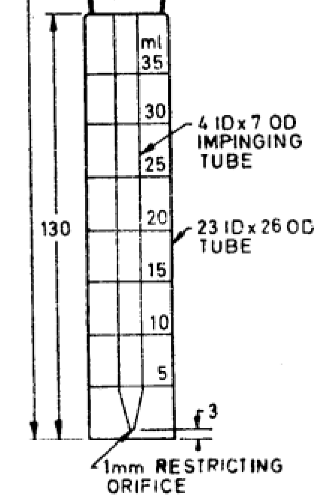

FIG.1 IMPINGER

7.1.1.6 *Timer and time totalizer*

The sampler shall have arrangement for run time display using a time totalizer with 1 sec resolution resettable/cumulative. It shall also have a 24 h programmable timer to automatically shut off the system after pre-set interval.

7.1.1.7 *Connecting tubing*

All tubing for interconnections shall be of inert silicon tube.

7.1.1.8 *Power supply and cords*

The system should work at 230 V a.c and power cords for supplying the power from the plug to the machine should be provided. The cabinet can also have an on/off switch.

7.1.2 The dimensions, finish and material of the cabinet and housing of the items in cabinet is at manufacturers discretion other than as defined in **7.1** but to ensure that it is sturdy, compact and light weight for outdoor field use as well as access or tempering is restricted. The cabinet can have pedestals to hold it stable on surface it is placed. The placings of the items be done in a manner that is practical and easy to use.

7.2 Spectrophotometer

A spectrophotometer suitable for measurement of absorbance at 560 nm with an effective spectral band width of not more than 5 nm is required. Problems may occur with spectrophotometer having greater spectral band widths. The wavelength calibration of the instrument shall be verified. If transmittance (*T*) is measured, this can be converted to absorbance (*A*) by the formula:

$$A = Log_{10}(1/T) \qquad \text{......(1)}$$

7.3 Glassware

Borosilicate and low actinic (amber colour) glassware or plasticware of equivalent quality as applicable.

7.4 Balance

An analytical balance with readability of 0.1 mg or better. The installation of balance shall be as per manufacturer's instruction on a vibration dampening surface.

8 REAGENTS

Quality of all reagents shall be analytical reagent grade, unless specified otherwise.

8.1 Reagent Grade Water

Reagent grade water of grade 2 minimum as per IS 1070 shall be used.

8.2 Potassium Tetrachloromercurate (TCM), 0.01 M

Dissolve 2.715 g mercuric chloride, 0.066 g EDTA, and 6.0 g potassium chloride in reagent grade water and bring to the mark in a 1 litre volumetric flask. The pH of this reagent shall be approximately 4.0 but it has been shown that there is no appreciable difference in collection efficiency over the range of *p*H 3 to *p*H 5. The absorbing reagent is normally stable for six months. If precipitate forms, discard the reagent after recovering the mercury.

NOTE — Highly poisonous if spilled on skin. Flush off with excess of water immediately.

8.3 Sulphamic Acid, 0.6 Percent

Dissolve 0.6 g of sulphamic acid in 100 ml of reagent grade water. Use within 10 days from the date of preparation

8.4 Formaldehyde, 0.2 Percent

Dilute 0.5 ml of formaldehyde solution (38 ± 2) percent to 100 ml with reagent grade water. Prepare fresh, when needed.

8.5 Stock Iodine Solution, 0.1 N

Take 6.35 g of iodine in a 250 ml beaker; add 20 g of potassium iodide and 25 ml of water. Stir to dissolve completely, then dilute to 500 ml with reagent grade water.

8.6 Iodine Solution, 0.01 N

Prepare approximately 0.01 N iodine solution by diluting 50 ml of stock solution (*see* **8.5**) to 500 ml with reagent grade water

8.7 Starch Indicator Solution

Triturate 0.4 g of soluble starch and 0.002 g of mercuric iodide preservative with a little water and add the paste slowly to 200 ml boiling water. Continue boiling until the solution is clear, cool, and

transfer to a glass-stoppered bottle.

8.8 Stock Sodium Thiosulphate Solution, 0.1 N

8.8.1 Prepare a stock solution by taking 25 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) in a beaker, add 0.1 g of sodium carbonate, and dissolve using reagent grade water (freshly boiled and cooled) making the solution up to a final volume of 1 litre. Allow the solution to stand one day before standardizing.

8.8.2 To standardize, accurately weigh, to the nearest 0.1 mg, 1.5 g of potassium iodate (KIO_3) certified reference material dried at 180 °C, dissolve, and dilute to 500 ml in a volumetric flask. Pipette 50 ml of the potassium iodate solution (0.1 N) into a 250 ml stoppered conical flask. Then add 2 g potassium iodide and 10 ml (1 : 10) hydrochloric acid to the flask and immediately stopper it. After 5 min, titrate with the stock thiosulphate solution to a pale yellow (straw) color. Add a few drops of starch solution and continue titration until the blue colour disappears. Note the titre value. Calculate the strength of the stock thiosulphate solution using normality equation:

$$N_1V_1 = N_2V_2 \quad \text{.…(2)}$$

where

V_1 = Volume of potassium iodate solution taken, that is, 50 ml;
N_1 = Strength of potassium iodate solution, that is, 0.1 N;
V_2 = Volume of thio-sulphate solution consumed (titre value); and
N_2 = Strength of the stock-thiosulphate solution.

NOTE — Certified reference material with certified value and associated uncertainty provided by an ISO 17034 accredited reference material producer with stated metrological traceability to SI units

8.9 Sodium Thiosulphate Titrant, 0.01 N

Dilute calculated ml of the stock thiosulphate solution to 1 litre with freshly boiled and cooled reagent grade water to get strength of solution 0.01 N.

Using normality equation:

$$N_1V_1 = N_2V_2 \quad \text{….(3)}$$

where

V_1 = Calculated volume of thiosulphate solution to be taken for dilution, in ml;
N_1 = Strength of Stock thiosulphate solution, as calculated in **8.8.2**;
V_2 = Volume of thiosulphate solution to be made that is 1 000 ml; and
N_2 = Strength of the stock-thiosulphate solution required that is 0.01 N.

8.10 Standard Stock Sulphite Solution

Dissolve 0.30 g of sodium metabisulphite ($Na_2S_2O_5$) or 0.40 g of sodium sulphite (Na_2SO_3) in 500 ml of freshly boiled and cooled reagent grade water. Sulphite solution is unstable must be prepared fresh for use. This solution contains the SO_2 equivalent of 320 µg/ml to 400 µg/ml.

The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulphate solution. To back-titrate, pipette out 50 ml of the 0.01 N iodine solution into each of two 500 ml iodine flasks A (blank) and B (sulphite solution). To flask A, add 25 ml of reagent grade water and into flask B, add 25 ml of sulphite solution. Stopper the flasks and allow to react for 5 min. By means of a burette containing standardized 0.01 N thiosulphate, titrate each flask in turn, to a pale-yellow color. Then add 5 ml of starch solution and continue the titration until the blue color disappears.

8.11 Working Sulphite — Tetra chloro mercurate (TCM) Solution.

Measure 2 ml of the standard sulphite solution (*see* **8.10**) into 100 ml volumetric flask by pipette and bring to mark with 0.01 M TCM (*see* **8.2**). Calculate the concentration of sulphur dioxide in the working solution in microgram of sulphur dioxide per ml. This solution is stable for 30 days if kept in the refrigerator at 5 °C. Otherwise, prepare fresh, when needed.

8.11.1 *Calculation*

Concentration of SO_2 solution, µg/ml =

$$\frac{(V_1\text{-}V_2) \times N \times 32\,000 \times 0.02}{25} \quad \text{…(4)}$$

where

C = Concentration of SO_2 solution, in µg/ml;
V_1 = Volume of thiosulphate used for flask A, in ml;
V_2 = Volume of thiosulphate used for flask B, in ml;
N = Normality of thiosulphate;

32 000 = Milli equivalent weight SO_2, in µg;
25 = Volume of standard sulphite solution, in ml; and
0.02 = Dilution factor.

Dilute sufficient volume of this known concentration of SO_2 solution (µg/ml) to prepare 100 ml standard solution with concentration of SO_2 2 µg/ml. Calculation can be done using standard equation:

$$N_1V_1 = N_2V_2 \qquad \text{.....(5)}$$

where

N_1 = calculated concentration of SO_2 solution, in µg/ml;
V_1 = required volume of working sulphite solution for dilution, in ml;
N_2 = 2 µg/ml SO_2 strength needed for standard solution; and
V_2 = volume of standard solution to be made i.e. 100 ml.

8.12 Preparation of Stock Pararosaniline Hydrochloride Solution, 0.2 percent concentration.

8.12.1 *Quality Assurance of Dye*

The pararosaniline hydrochloride dye shall have a wavelength of maximum absorbance at 540 nm when assayed in a buffered solution of 0.1 M sodium acetate-acetic acid; the absorbance of the reagent blank which is temperature-sensitive to the extent of 0.015 absorbance unit/°C, shall not exceed 0.170 absorbance unit at 22 °C with a 1 cm optical path length, when the blank is prepared according to the prescribed analytical procedure and to the specified concentration of the dye; the calibration curve, shall have a slope of (0.030 ± 0.002) absorbance unit/µg SO_2 at this path length when the dye is pure and the sulphite solution is properly standardized.

8.12.2 *Preparation of Stock Solution of Pararosaniline Hydrochloride,* (0.5 percent)

Dissolve 0.5 g of pararosaniline hydrochloride in 100 ml reagent grade water. Keep it for 2 days and filter the solution using any ashless (≤ 0.007 percent) filter paper with pore size in the range 20 µm to 25 µm. The filtrate solution is stable for 3 months, if stored in the refrigerator at 5 °C or less.

8.12.3 *Working Pararosaniline Hydrochloride Solution,* (0.02 percent)

Add 15 ml concentrated hydrochloric acid to 10 ml stock pararosaniline solution and dilute with reagent grade water in a 250 ml volumetric flask up to the mark. This solution is light greenish yellowish in colour. It is stable maximum up to two weeks when stored in the refrigerator at 5 °C or less in an amber colored bottle.

9 SAMPLING

The sampling for SO_2 is specified in Table 1.

9.1 Location of Sampling Station/Sampler

For planning of network of sampling stations including number of stations and location of sampling station or sampler refer IS 5182 (Part 14). Record topographical features, sources of pollution, any obstructions to sampling station including any deviations specific to site and with relevance to objectives of monitoring on the sampling sheet.

9.2 The sample shall be drawn as specified in Table 2.

Table 1 Sampling for SO_2
(*Clause* 9)

Sl No.	Pollutant	Time Weighted Average	Concentration in Ambient Air	
			Industrial, Residential, Rural and Other Area	Ecologically Sensitive Area (Notified by Central Government)
(1)	(2)	(3)	(4)	(5)
i)	Sulphur Dioxide (SO_2) µg/m^3, (micrograms per cubic meter of air)	Annual 24 h	50 80	20 80

NOTE — One can select other combinations of duration, period and frequency to meet objectives of the sampling with corresponding adjustments in flow rate and absorbing solution in impinger to maintain linearity between absorbance and concentration.

Table 2 Frequency of Drawing Sample
(*Clause* 9.2)

Sl No.	Duration	Period	Frequency	Flow Rate, litre/min	Absorbing Solution in Impinger, in ml
(1)	(2)	(3)	(4)	(5)	(6)
i)	24 h	24 h	Single	0.5	30
ii)		8 h	3 Shifts	1	30
iii)		4 h	6 Shifts	1	30
iv)	Annual	24 h	104 Measurements for 24 h Single run or 8 h × 3 shifts or 4 h × 6 shifts	0.5 for single run; 1 for 8 h × 3 shifts or 4 h × 6 shifts	30

NOTES

1 Annual arithmetic mean of minimum 104 measurements in a year at a particular site taken twice a week 24 hly at uniform intervals.

2 Since practically sometimes it may not be possible to target sampling of 24 h in a day or 104 measurements in a year due to power failure or other site-specific problems, the values monitored for 75 percent of the time and more can be considered as representative SO_2 values in the ambient air.

9.3 Operation of Sampler

The manufacturer's instructions are to be followed.

The sampling shall be done as follows:

a) Carry requisite quantity of absorbing solutions to sampling site in chemical resistant HDPE bottles with leak-proof polypropylene screw cap meant for the purpose of packing, shipping and storage in insulated box;

b) Place standard 35 ml midget impinger in gas sampler. Impingers shall be cleaned before each sampling to remove deposited chemicals on surfaces and clogging chemical residues at impinger midget tube. To make impinger leak proof silicon grease must be applied at joint of impinge;

c) Add 30 ml TCM solution to the impinge;

d) Run sampler for such period and frequency as prescribed in the standard;

e) Record initial time totalizer reading;

f) Switch on the sampler;

g) Record initial flow rate;

h) Monitor flow rate during sampling. For 4 h run initial and final flow rate may suffice. For 8 h or 24 h single run record flow rate at regular intervals. If flow rate varies from set value by more than 15 percent stop the sampling. The sampler may need service and recalibration before using again;

j) At the end of sampling period and frequency record final flow rate;

k) Switch off the sampler;

m) Record final time totalizer reading;

n) Record reasons any loss of time due to power failure or any other malfunctions;

p) Check loss of water from absorbing reagent in impinger and make it up using reagent grade water Grade-2) before transferring it into sample storage bottle at site;

q) Transfer sample storage bottle to lab in insulated box; and

r) In laboratory, store the sample in a refrigerator at 5° C or less. When samples are stored at 5° C for 30 days, no detectable loss of sulphur dioxide occurs.

NOTES

1 The presence of EDTA in absorbing solution enhances the stability of SO_2 in solution. The rate of decay is independent of the concentration of SO_2. At 22 °C losses of SO_2 occur at the rate of 1 percent per day. In hot weather when temperature exceeds 30 °C, maintain (20 ± 5) °C temperature in insulated box during transportation using ice/chilled water.

2 Concentration of SO_2 in ambient air may have an impact depending upon meteorological conditions as temperature, relative humidity, rainfall on the day of sampling. The wind speed and mixing height data can also influence the level of concentrations.

10 PROCEDURE

10.1 Check sample before analysis for any precipitates. If any precipitate is observed remove it through centrifuging and analyze supernatant solution for SO_2.

10.2 Delay analysis for 20 min to allow ozone (if any) to decompose before analysis. This is required if sample is being taken directly from sampler to laboratory for immediate analysis.

10.3 For each set of determinations prepare a (I) reagent blank by adding 10 ml of unexposed TCM solution to a 25 ml volumetric flask, (II) prepare a control solution by measuring 2 ml of working sulphite — Tetra chloro mercurate (TCM) Solution into a 25 ml volumetric flask by pipette and (III) take 10 ml aliquot of each sample to 25 ml volumetric flasks equal in number of samples to be analyzed.

10.4 To each volumetric flask containing sample or reagent blank or control solution add 1 ml of 0.6 percent sulphamic acid and allow to react for 10 min to destroy the nitrite ion resulting from oxides of nitrogen. Add 2 ml of 0.2 percent formaldehyde solution and 2 ml of para- rosaniline solution in order. Start a laboratory timer that has been set for 30 minutes. Bring all volumetric flasks to 25 ml mark with freshly boiled and cooled reagent grade water (Grade 2) and mix thoroughly. Within 30 min to 60 min, determine the absorbance of the reagent blank, control solution and sample at 560 nm using cells with a 1 cm path length. Use reagent grade water (not the reagent blank) as the optical reference. This is important because of the color sensitivity of the reagent blank to temperature changes which may be induced in the cell compartment of a spectrophotometer.

If the absorbance of the sample solution lies between 1.0 and 2.0 the sample solution can be diluted to 1 : 1 with a portion of the reagent blank and read within a few minutes. Solutions with higher absorbance can be diluted up to six-fold with the reagent blank to obtain on-scale readings within 10 percent of the true absorbance value

NOTE — Do not allow the colored solution to stand in the absorbance cells of spectrophotometer, because a film odye may be deposited. Clean cells with the help of alcohol and

cleaning reagent immediately after use.

10.5 *Calibration Curve*

Measure 0 (blank), 0.5 ml, 1 ml, 1.5 ml, 2 ml, 2.5 ml, 3 ml, 3.5 ml and 4 ml of working sodium sulphite-TCM solution into a series of 25 ml volumetric flasks. Add sufficient TCM solution to each flask to bring the volume to approximately 10 ml. Then add the remaining reagents as described in **4.7.4.** Read the absorbance of each standard solution against reagent grade water. Plot a curve absorbance (Y axis) versus concentration (X axis). Draw a line of best fit and determine the slope. The reciprocal of slope gives the calibration factor (CF). This calibration factor can be used for calculating results provided there are no radical changes in temperature or *p*H. A control containing a known concentration is recommended to ensure the reliability of this factor

For maximum precision use a constant temperature bath. The temperature of calibration shall be maintained within 1°C and within the range of 20 °C to 30 °C. The temperature of calibration and that of analysis shall be maintained within 2 °C. If the temperature of the determinations does not differ by more than 2 °C from the calibration temperature, the reagent blank should be within 0.03 absorbance unit of the y-intercept of the calibration curve. If the reagent blank differs by more than 0.03 absorbance unit found in the calibration curve, prepare a new curve to minimize error.

11 CALCULATION

For calculation of SO_2 in ambient air use following formula

$$\text{Concentration } SO_2, \mu g/m^3 = \frac{(A_s - A_b) \times CF \times 1\,000 \times V_{abs} \times D}{V \times V_{test}} \quad \ldots(6)$$

where

A_s = Absorbance of sample;
A_b = Absorbance blank;
CF = Calibration factor;
1 000 = Conversion of litre to m^3;
V_{abs} = Volume of absorbing solution taken in impinger while sampling (30 ml), in ml;
V_{test} = Volume of sample taken for analysis (10 ml), in ml; and
D = Dilution factor.

The concentration of sulphur dioxide may be calculated as parts per million of sulphur dioxide at reference condition as given in equation

SO_2 ppm = µg SO_2 /m^3 × 3.82 × 10^{-4} (that is 1 ppm SO_2 = 2 618 µg /m^3 SO_2 at 25 °C and 760 mm Hg pressure) ...(7)

12 QUALITY ASSURANCE/QUALITY CONTROL MEASURES

12.1 Calibration

Periodic calibration of the gas sampler shall be performed as per the calibration plan prepared by laboratory at such intervals as determined based on use and subject to performance of intermediate checks at regular intervals for flow to maintain confidence in the performance of sampler. The acceptance criteria for flow rate shall be ± 5 percent. Laboratory shall maintain a master calibrator for doing intermediate checks inhouse.

12.2 Field Blank

Purpose of field blank is to detect and identify any contaminant from the sampling site including travelling to and from the laboratory to the site. For field blanks, impinger with absorbing solution is placed in the sampler for the sampling duration. The air is not bubbled through the impingers. By doing this, the field blanks are exposed to the atmosphere of the sampling site and any contamination due to handling. The absorbing solution is analyzed for any concentrations of interest.

12.3 Leak Check Procedure and Criteria

Follow the sampler manufacturer's manual to do the leak check (**7.1.1.4**) which must meet predefined leak check acceptance criteria. Create 200 mm Hg vacuum in the complete sampling system including inlet/outlet tubing with many fold, valves and impinger. Vacuum must not reduce more than 25 mm Hg in 3 min.

12.4 Proficiency Testing

Laboratories shall participate in proficiency testing programs as per the Proficiency Testing plan prepared by laboratory.

13 PRECISION AND BIAS

Relative standard deviation at the 95 percent confidence level is 5 percent for the analytical procedure using standard sample.

ANNEX A
(*Foreword*)

COMMITTEE COMPOSITION
Air Quality Sectional Committee, CHD 35

Organization	*Representative(s)*
In Personal Capacity (*23E/202, Palazzio CHS, S. Marg, Powai, Mumbai 400 076*))	DR GAURI PANDIT (***Chairperson***)
Bhabha Atomic Research Centre, Mumbai	DR A. VINOD KUMAR DR S. K. SAHU (*Alternate*)
Central Institute of Mining and Fuel Research, Dhanbad	DR R. C. TRIPATHI DR N. K. SRIVASTAVA (*Alternate*)
Central Pollution Control Board (MoEFCC), GoI, New Delhi	SHRI V. K. SHUKLA DR R. C. SRIVASTAVA (*Alternate*)
Confederation of Indian Industry, New Delhi	DR SANTOSH KUMAR YADAV MS ANJALI (*Alternate*)
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In formulation of this standard, the support extended by following laboratories in doing field studies to establish reduction in use of mercury in preparing TCM solution as per laboratory work done by CSIR-NEERI Delhi is gratefully acknowledged-

a) MCF Environment Laboratory, Mangalore Chemicals & Fertilizers Limited, Karnataka;
b) Central Laboratory, Madhya Pradesh Pollution Control Board, Bhopal, Madhya Pradesh;
c) Gujarat Environment Management Institute, Ahmedabad, Gujarat;
d) EMTRC Consultants Pvt Ltd, Ghaziabad, Uttar Pradesh;
e) Siddhi Green Excellence Private Limited, Ankleshwar, Gujarat;
f) 10, Saharanpur, Uttar Pradesh; and
g) Central Laboratory, Telangana State Pollution Control Board.

The composition of the committee responsible for formulation of this standard is listed Annex A.

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This Indian Standard has been developed from Doc No.: CHD 35 (18812).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi